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Improving fibrillation and structural integrity of coconut frond fibers with alkali and steam processing

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ABSTRACT

The effects of alkali treatment steaming on the fibrillation of coconut frond stem fibers has been successfully evaluated. The applied procedures include physical and chemical treatments to reduce the fiber size from the micrometer to the nanometer scale. The fibers underwent alkali treatment using NaOH solutions at varying concentrations, followed by steaming to facilitate fiber separation. The results indicate that a 2 % NaOH solution with a 4-hour immersion produced the highest tensile strength, reaching 276 MPa, compared to untreated fibers, which exhibited a tensile strength of 155 MPa. FTIR and SEM analyses revealed the effectiveness of these treatments in breaking lignin and hemicellulose bonds and increasing fiber crystallinity. The combination of chemical and physical treatments significantly enhances the mechanical properties and microstructure of coconut frond fibers, presenting new opportunities for sustainable material applications.

1. Introduction

The study of natural sources and fibers in both macro and micro scales now needs to be sharpened to reduce the limitations of mechanical properties and their applications [1,2]. Natural fibers are classified into plant-based, animal-based, and mineral-based fibers. Plant-based fibers consist of cellulose, hemicellulose, and lignin. These components determine its strength, flexibility, and thermal properties, which are influenced by factors such as geographical location, extraction method, and the specific plant part used. The fibers are proven can be applied in different applications i.e. automotives and structures [3,4]. The initial physical treatment for the cellulose fibrillation process was carried out by adjusting the fiber dimensions in which the length of the natural fibers prior to entering the pressurized reactor [5]. Furthermore, other techniques are demonstrated [6] through the spraying of water vapor temperature of 100 °C–150 °C in the fibers of coconut fiber (coir) before being subjected to high pressure steam. Steam treatment applications for breaking down natural fibers are also generated through explosion and refining processes. This treatment increases fiber crystallinity and tensile strength by removing amorphous components such as lignin and hemicellulose, as evidenced by studies on fibers such as flax and pineapple that show an improvement in fracture strength [3].

Initial chemical treatments for the fibrillation/microfibril cellulose process were carried out with several chemicals including NaOH, acetic acid, sulfuric acid, sodium hypochlorite, oxalic acid [7]. Alkali treatment has been extensively studied for its effectiveness in removing impurities, increasing cellulose content, and improving mechanical properties [3]. Another study used initial treatment of Salix miyabeana fiber by immersion mechanism in sulfuric acid solution of 3 % composition (wt.%) For 10 min [8]. Furthermore, the strengthening of natural fibers by chemical treatment was carried out by immersion / dipping into solution containing hydroxide (OH) groups, silicon (Si), chloride (Cl) and other fiber coating groups accompanied by treatment of the process conditions. Advances in the treatment of these fibers have the potential for use in automotive components, eco-friendly packaging, and lightweight construction panels [9]. Future studies could explore the

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integration of natural fibers treated with biodegradable polymers to create fully sustainable composites. Additionally, investigating the environmental stability of treated fibers under varying conditions will optimize their performance for diverse applications.

2. Material and method

2.1. Material

The primary material used in this study is coconut frond stem fibers. Sodium hydroxide (NaOH) was used for the alkali treatment, and distilled water was used to prepare the solutions. The coconut fronds were collected locally, while all chemicals were sourced from commercial suppliers. Equipment used for the experiments included cutting tools, a roller machine for softening the fibers, and an oven for drying. Analytical equipment included a tensile strength tester, Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and X-ray Diffraction (XRD) for characterizing the fibers.

2.2. Methods

The coconut frond fibers were first cut into 50 cm sections and split into smaller pieces. These pieces were softened with a roller machine and cleaned manually to remove wax, lignin, and hemicellulose. After cleaning, the fibers were dried at 80 °C for 24 h to remove moisture. Next, the dried fibers were treated with sodium hydroxide (NaOH) solutions at concentrations of 1 %, 2 %, 3 %, and 4 %. The selected durations and concentrations were based on findings that short-term treatments with lower NaOH concentrations effectively delignify fibers without causing excessive structural degradation [10]. Previous studies indicate that longer durations or higher concentrations can lead to fiber damage and reduced mechanical properties.

The fibers were soaked in NaOH for 30 min at room temperature, then washed thoroughly with distilled water to remove any remaining NaOH. They were dried again at 80 °C for another 24 h. Following this, the fibers underwent steam treatment at atmospheric pressure and 100 °C for 1 h, then dried again at 80 °C for 24 h to ensure complete drying. To assess the fibers' mechanical and chemical properties, several tests were performed. Tensile strength was measured using ASTM D3379 (2016) standards. FTIR analysis was used to examine chemical changes, SEM to observe surface morphology, and XRD to measure crystallinity and detect structural changes in cellulose after treatment.

3. Result and discussion

3.1. Tensile strength result

Tensile strength that shown in Fig. 1 indicate that coconut frond fibers treated with 2 % NaOH for 4 h achieved a maximum strength of 276 MPa, compared to 155 MPa for untreated fibers. This improvement is attributed to the partial removal of lignin and hemicellulose, which allows better alignment of cellulose fibrils and enhances fiber–matrix bonding in composite applications. Strength increased as NaOH concentration rose, peaking at 2 %. However, higher concentrations weakened the fibers due to excessive removal of lignin and hemicellulose, leading to structural degradation. This outcome aligns with findings on bamboo fibers, where alkali treatments enhanced tensile strength and crystallinity but excessive treatments led to over-degradation [11]. Treatment with 2 % NaOH for 4 h was found to be optimal for enhancing the mechanical properties of coconut frond fibers.

3.2. FTIR Result

FTIR analysis that illustrated in Fig. 2 results for untreated and NaOH-treated coconut frond fibers showed peaks for key components: cellulose, hemicellulose, and lignin. Peaks at 3425 cm^{-1}, 2924 cm^{-1},

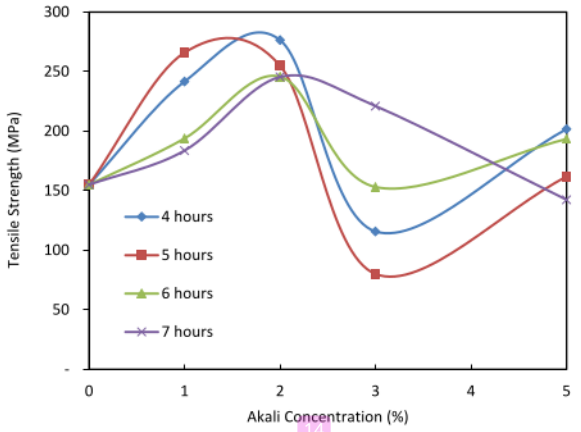

Fig. 1. Effect of concentration and time of alkali treatment on tensile strength of bundle fibers.

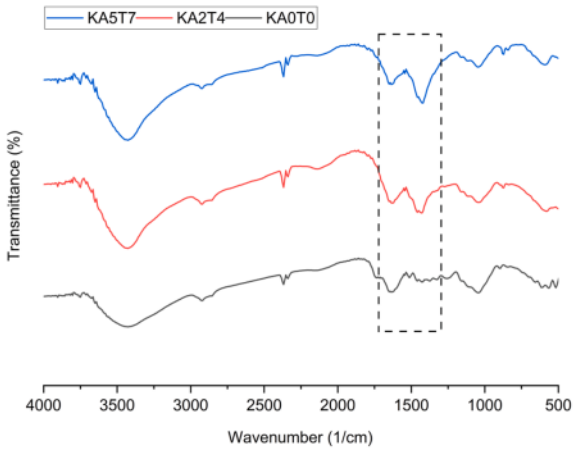

Fig. 2. FTIR test results.

and 1720 cm^{-1} appeared in untreated fibers, representing O—H stretching (hydroxyl groups in cellulose), C—H stretching (methyl and methylene groups), and C═O stretching (ester groups in hemicellulose), respectively. After treatment with 2 % NaOH, the peaks at 1720 cm^{-1} and 2924 cm^{-1} reduced in intensity, indicating the removal of hemicellulose and lignin. The peak at 3425 cm^{-1} became more pronounced, suggesting improved hydrogen bonding within cellulose fibrils. These changes are consistent with findings in [12] where alkali treatments reduced non-cellulosic components and enhanced hydrogen bonding, contributing to tensile strength improvement.

3.3. SEM Result

SEM observations revealed clear differences in the surface morphology of untreated and treated fibers as seen in Fig. 3(a-b). Untreated fibers (KA0T0) that shown in Fig. 3(a) exhibited a smooth surface with tightly bound layers, indicating the presence of lignin and hemicellulose as binding agents. In contrast, treated fibers (KA2T4) showed a rougher, more porous surface with distinct separations between fiber layers (Fig. 3(b)). The diameter of the fibers decreased from 120 μm (untreated) to 95 μm (treated) reflecting the removal of non-cellulosic materials and compaction of cellulose fibrils. Similar

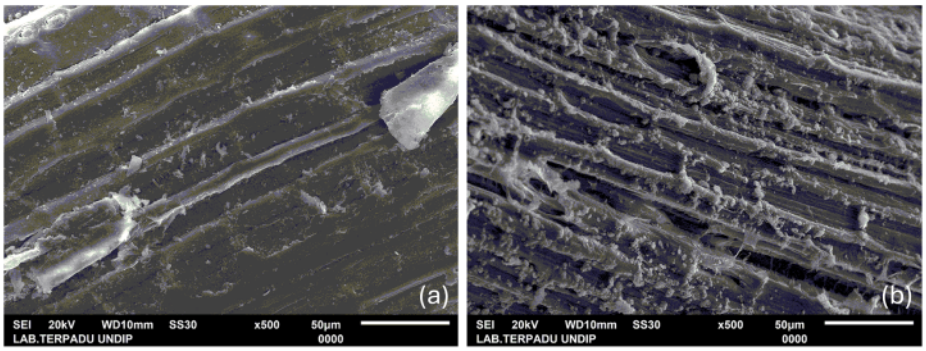

Fig. 3. SEM observation of KA0T0 specimens (a) and KA2T4 specimens (b).

findings were reported for bamboo and Palmyra fibers, where alkali treatment resulted in enhanced fibrillation and improved mechanical interlocking in composites [11,12]. The increased porosity and fibrillation observed in the SEM images contribute to better interfacial bonding and mechanical properties, as evidenced by the tensile strength results.

3.4. XRD Result

XRD analysis showed a significant increase in the crystallinity index of coconut frond fibers after alkali and steam treatment as shown in Fig. 4. The crystallinity index (CI) increased from 66 % (untreated fibers) to 76 % (treated fibers). This improvement is attributed to the removal of amorphous components, such as lignin and hemicellulose, which makes the crystalline cellulose regions more prominent. This finding is consistent with studies on bamboo fibers where alkali treatments improved crystallinity and thermal stability and enhancing mechanical properties [11]. The XRD graph was redrawn starting from 0° to provide a complete visualization of the crystalline and amorphous regions. This approach ensures a more accurate calculation of the crystallinity index and highlights the structural changes induced by the treatment.

4. Conclusion

This study demonstrated that the combination of alkali treatment with 2 % NaOH for 4 h and subsequent steaming significantly enhanced the properties of coconut frond fibers. The tensile strength increased to 276 MPa, nearly doubling the strength of untreated fibers (155 MPa). FTIR analysis confirmed the removal of lignin and hemicellulose, SEM observations revealed a reduction in fiber diameter from 120 µm to 95 µm, and XRD analysis indicated an increase in crystallinity index from 66 % to 76 %. These results suggest that coconut frond fibers have great potential as eco-friendly reinforcement materials for composite applications.

CRediT authorship contribution statement

Seno Darmanto: Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Rahmad Kuncoro Adi:** Writing – review & editing, Writing – original draft, Visualization. **Djarot Widagdo:** Writing – review & editing. **Gil Nonato C. Santos:** . **Mohd. Ridwan:** Writing – review & editing. **Muhammad Akhsin Muflikhun:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Funding acquisition.

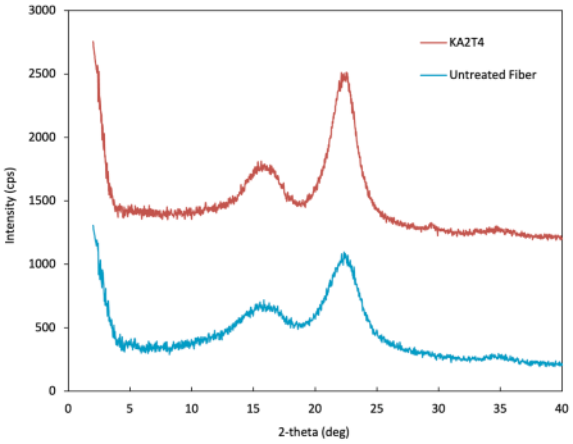

Fig. 4. XRD test results.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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